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Final Report			· ·	
Denny F. Brown Charles T. Derick				
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The development and pilot production of an improved blister sheet and pouched overwrapper package for the icdine water purification tablet is described. The blister sheets are fabricated from a fluorohalocarbon film containing 12 tablets with each tablet isolated by a heat sealed gridwork. A pressure sensitive label adheres to each blister sheet. This label is fabricated so that use instructions are on one side and a tablet gray color match is on the adhesive side to act as a tablet potency indicator. The pouch overwrapper is fabricated from a laminated film of Mylar-aluminum foil polyolefin and is designed to contain two blister sheets. Three major shortcomings of the initial flexible package have been corrected. The excessive amount of moisture entrapped within the labels when the blister sheets were sealed in the pouch has been eliminated by predrying the labeled blister sheets in a desiccant prior to pouching. Weak heat seals existing around corner blisters have been corrected by redesigning the blister sheet heat seal ing tray to include larger sealing elements, installation of legs on the lower half of the tray for localized pressure application, and addition of ventilation holes to dispel residual heat buildup. High production cost primarily resulting from excessive fluorohalocarbon film scrap rate has been eliminated by modifying blister forming mold.

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DEVELOPMENT OF AN IMPROVED BLISTER SHEET AND POUCHED OVERWRAPPER PACKAGE FOR IODINE WATER PURIFICATION TABLETS

Final Report Contract No. DAAD03-72-C-0028

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<u>ACKNOWLEDGMENTS</u>

The authors appreciate the technical assistance and guidance furnished by Mr. Joseph Carney of the U. S. Army Land Warfare Laboratory during the design and production phases of this project. In addition, Mr. Dennis Hurley of Columbia Research Corporation is to be recognized for his extensive participation in all of the production operations required for producing the required 30,000 units of the improved flexible package.

SUMMARY

Improvements in the design, techniques of manufacturing, and procedures used in producing a pilot production of 30,000 experimental flexible packages containing iodine water purification tablets are presented herein. The work has been performed under a U.S. Army contract. The package consists of a pouch in which two blister sheets are sealed. Each of the blister sheets contains 12 tablets which are isolated from one another by a heat-sealed gridwork. The label applied to the back of the blister sheet has the use instructions and is colored gray on its adhesive side to match the color of a potent tablet. The gray color provides a matching background around the tablet when the blister sheet is viewed from the front. Because degradation of the tablet is reflected by a change in its color, this feature permits the user in the field to easily discard those tablets which have deteriorated.

An initial effort to produce a pilot production of the flexible package on a previous contract revealed certain deficiencies which have since been corrected. The major shortcoming of the original package was discovered to be an excessive amount of moisture entrapped within the label when the blister sheets were sealed in the pouch. When the package was subjected to accelerated storage testing at elevated temperature, this moisture was released within the pouch and submitted the blister sheets to a hot-humid atmosphere. The humidity then permeated the blister sheet film and destroyed the tablets. This problem has since been remedied by pre-drying the labeled blister sheets in a desiccant of calcium sulfate for 1 week prior to pouching. A repeat of the accelerated environmental tests using new packages made with predried labels has shown them to exhibit less deterioration than 50-tablet bottles (current Army package) submitted to the same tests.

A second problem associated with the original flexible package was that weak heat seals existed around corner blisters and resulted in leaks during vacuum leakage tests. This problem has been corrected by a redesign of the blister sheet heat sealing tray. Changes include replacement of the sealing elements with replaceable sealing bands that increase the seal width from 3/32" to 1/8"; installment of legs on the lower half of the tray to apply localized tray pressure to counteract the effects of tray warpage; and the addition of ventilation holes and spaces in the upper half of the tray to dispel the residual heat buildup occurring in production. The result of all these modifications has led to the production of new blister sheets which have tighter, more uniform seals.

A third problem of the original flexible package was its high production cost resulting primarily from the excessive scrap rate of the Aclar 22A film used in the formation of blister sheets. By modification of the blister forming mold, the amount of film required to make blister sheets has been reduced by 32%, while adjustments to the blister forming machinery have increased the rate of production by 100%. In addition, the high waste associated with the fabrication of malformed blisters on the original production has been virtually eliminated. It is anticipated that this improvement, coupled with increased automation of the other processes using standard production methods, will result in a produced package at a cost comparable to the 50-tablet bottle.

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INTRODUCTION

The present packaging container for the iodine water purification tablets (each tablet containing 17 percent tetraglycine by droperiodide as the active ingredient) consists of a small 50-cablet bottle. Primary closure of the bottle is by means of a screw cap having a vinyl coated liner. The secondary closure is a wax coating to seal the juncture between the cap and bottle. Once the wax seal is broken, the secondary closure is no longer effective. Each time the bottle is opened, both the partial pressure of the iodine vapor is decreased, and moisture is introduced, resulting in sublimation and deterioration of the remaining tablets. Deterioration of the tablets is more rapid in high temperature/high humidity environments such as in Southeast Asia. The iodine water purification tablets should therefore be protected against moisture absorption and loss of iodine vapor/partial pressure until time of use to ensure full potency of the tablets.

The task of improving the present method of packaging iodine tablets has been previously undertaken. The approach consisted of sealing the individual tablets in blister packs and strip packs made from various transparent packaging films. Of the various films evaluated, Aclar 22A (Allied Chemical Company) was the most acceptable material from the standpoint of being a relatively good barrier to iodine vapor. The blister pack concept, rather than the strip pack, was preferred because of its greater compactness for packing in individual survival kits or carrying in the pouch which is attached to the canteen covers currently in the Army supply system.

Columbia Research Corporation (CRC) undertook a development and pilot production effect under Contract No. DAADC5-70-C-0089 with the U. S. Army Land Warfare Laboratory (USALWL) to develop a new and improved package for the indine tablets. The new method of packaging consisted of sealing the individual tablets in blister sheets and packaging two blister sheets in an overwrapper packet. Each blister sheet, measuring 1-3/4" x 2-1/8", contained 12 transparent blister units on a steel gray color match background. background was used because a change in color indicates loss of potency. Two blister sheets of 12 tablets each were overwrapped for added protection. The new, simplified directions for use were printed on the back of each blister sheet and on the exterior surface of the overwrapper packet. The new package underwent 6 months of accelerated laboratory storage testing. As a result of these tests, it was concluded that although the proposed packaging concept and packaging materials were acceptable, the new package was unacceptable because of shortcomings in the packaging process2.

The U. S. Army Land Warfare Laboratory (USALWL) awarded a contract to Columbia Research Corporation (CRC) to make the necessary improvements to correct the original packaging deficiencies, and to fabricate another pilot production quantity of this package which again will be subjected to U. S. Army laboratory and field testing.

This report describes improvements made to fabrication equipment, evaluates the results of quality control tests, and describes and evaluates the manufacturing processes used in fabrication of the pilot production of 30,000 units.

IMPROVEMENT_OF FABRICATION EQUIPMENT AND ITEMS

Blister Forming Molds

The Government-furnished blister forming molds were designed for use with a Model JF-14 Packaging Industries, Inc. Blister Forming Machine. A machine of this type was used by CRC on this contract.

To reduce the production costs of blister sheets, the forming molds and associated equipment were modified. Modifications included machining 1 inch off the width of the base of the forming molds, and the fabrication of a new stripper plate and forming box to accommodate this change. Figure 1 shows the design drawing of one of two identical sections of the modified forming mold, while Figure 2 shows the dimensions of the blister sheet formed by these In addition, the JF-14 was adjusted to produce blisters from 14 1/2-inch wide rolls of Aclar 22A instead of the 15 1/2and 15 3/4-inch wide rolls which were used on the original contract. To eliminate the problem of film foldover when drawn into the forming mold, the feed chains that engage the film edges were spaced slightly wider apart at the film exit point than at the film entry point. This caused stretching of the film in the transverse direction as it was fed through the machine, and had the effect of taking up the excessive film sag occurring during the preheating phase.

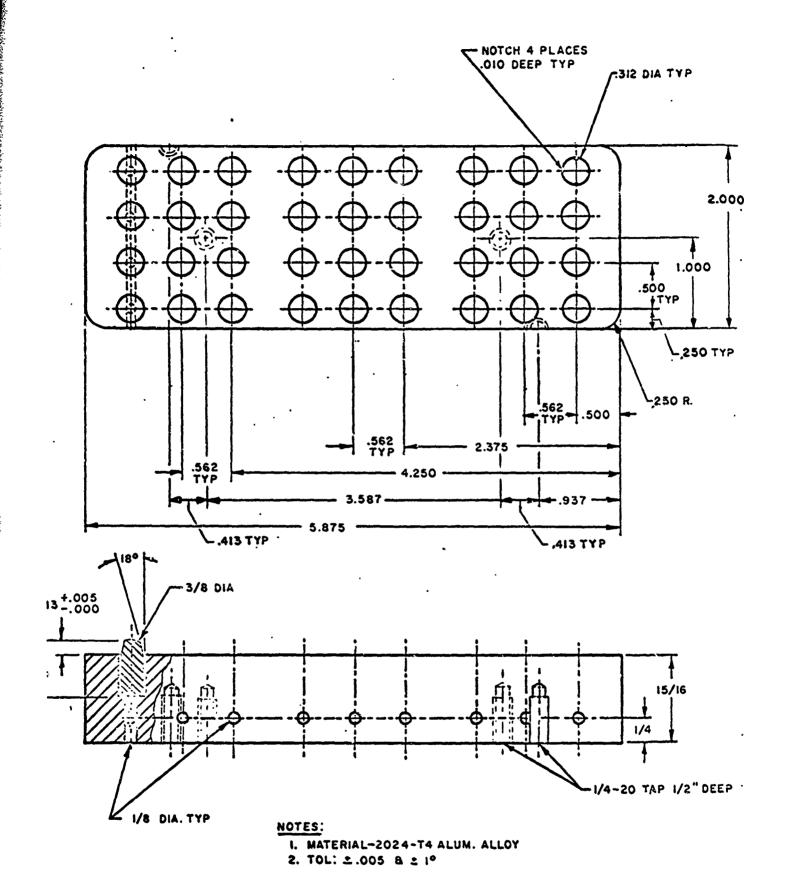
The results of the above improvements are tabulated below:

entropies of the contraction of

	<u>Original</u>	<u>Modified</u>
Film roll width	15-1/2" and 15-3/4"	14-1/2"
Size of formed blister sheet (6 doz. blisters)	3.562" x 13.094"	2.500" x 13.094"
Rate of production (cycle period)	8.0 sec.	4.1 sec.
Malformed blister sheets (percent)	30	5

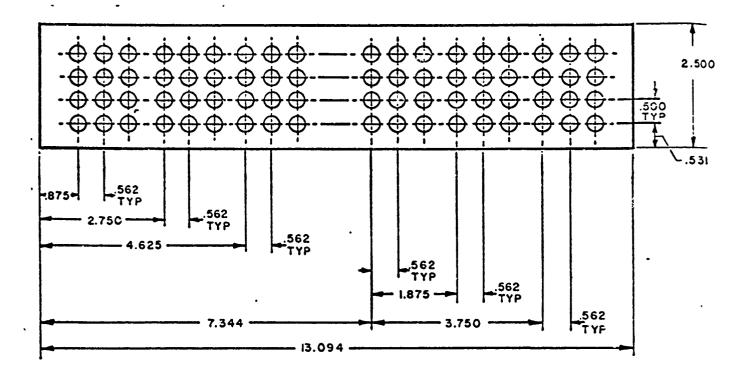
Sealing Tray

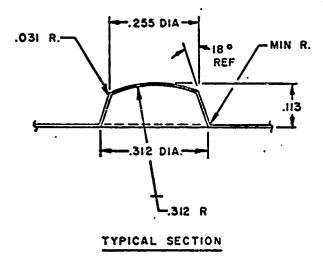
The Government-furnished sealing tray was designed for use on a Packaging Industries, Inc. (PI) F-20 Blister Heat Sealer. Attempts to use this tray as furnished produced seals having several hot spots, indicating that the heating elements were on the verge of burning out. This tray, even when new, was of marginal quality



的,我们是是一个人,我们是一个人的,我们是一个人的,我们是一个人的,我们是一个人的,我们也不会一个人的,我们也没有一个人的,我们也是一个人的人的,我们就是一个人的

FIGURE 1. MODIFIED BLISTER FORMING MOLD





NOTES

- I. MATERIAL: -. OOS THK. ACLAR
- 2. DIMENSIONS APPLY AFTER MOLDING
- 3. TOL: 2.015 8 ± 10

FIGURE 2. VACUUM FORMED BLISTER SHEET

since it produced seals at the corners of the uncut blister sheets which had a high rate of failure during vacuum leakage tests. Quality control tests had one or more bad seals. Accordingly, it was considered prudent to subcontract to PI the task of making the necessary repairs and design modifications to this tray so that it would produce uniform and reliable seals.

The major change in tray design by PI was to replace the heat sealing elements with replaceable stainless steel bands of increased width. These bands increased the width of the seals from 3/32" to 1/8". A second modification was the addition of a series of eight supporting legs on the lower half of the tray to permit adjustments of localized tray pressure as needed to offset any tray warpage. A third modification was to ventilate the upper tray with a series of holes and spacers to reduce the residual heat build-up during production sealing operations. These improvements have resulted in blister sheets with seals of high integrity and uniformity.

Cutting Die

The Government-furnished cutting die used to cut the blister sheets of six dozen tablets into six sheets of one dozen tablets each was dulled from use on the original pilot production. The cutting blades were removed, sharpened, and reinstalled to restore it to a good operational condition. Figure 3 shows a drawing of a typical die cut blister sheet.

Pouches

The Continental Can Corporation manufactured the pouch overwrappers for the blister sheets from a laminate film of .5 mil Mylar/.35 mil al foil/3 mil C-79 polyolefin; measuring 2-5/8" x 3-3/8". As with the previous contract, because of the small production quantity, they were made individually with three edges sealed for hand filling of the blister sheets. Also, because of the limited production order, they were again printed flexographically with the specified information and coloring externally on the Mylar. The edges were left bare to avoid ink removal during heat sealing. In high production quantities, the Mylar would be reverse printed on its inside face prior to lamination. This would also permit total coloring of the pouch including the seal areas.

The pouches, however, were modified in several aspects. Specifically, the bottom seal of the pouch was reduced in width from 1/4" to 3/16". This change allowed more space to permit a wider

SCALE 2"= |"

FIGURE 3. IODINE WATER PURIFICATION TABLET BLISTER SHEET

final seal with the Doughboy HS-B sealer when the pouch was filled with the blister sheets while maintaining the original overall dimensions. A second change in the design was to move the tear notch so that tearing would be accomplished along the width of the pouch. In addition, the purchase order to Continental Can required that their seals meet Requirement 2024 of Federal Test Method Standard No. 101B. This regulation states in essence that a 1-inch wide strip of the film containing the seal be able to withstand a 50 oz. static load for 5 minutes. Figure 4 shows the printing layout and configuration of the supplied pouches.

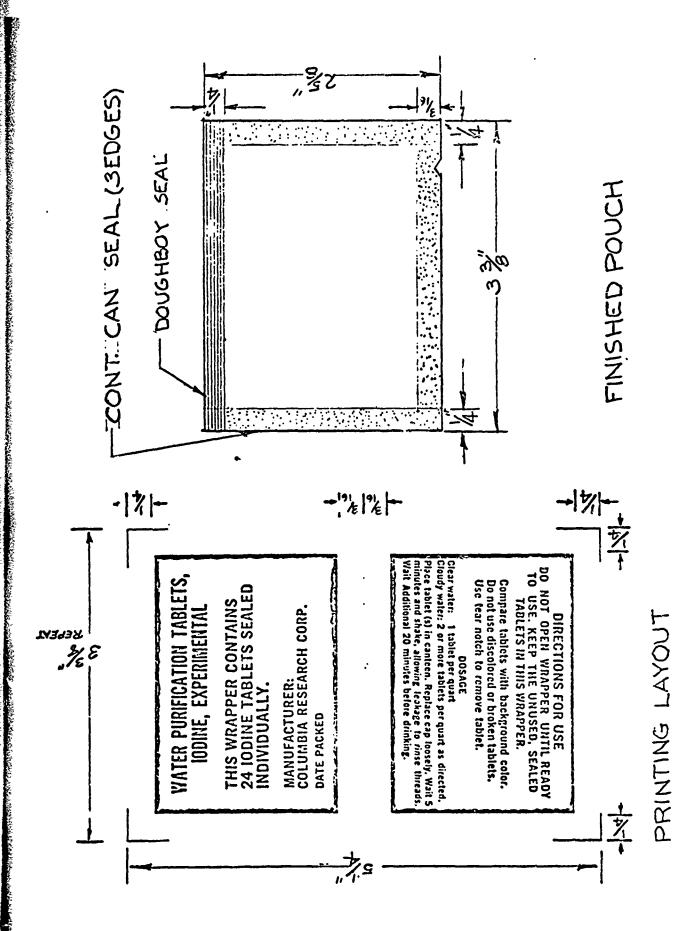


FIGURE 4. POUCH OVERWRAPPER

QUALITY CONTROL TESTS

General

The U. S. Army supplied the iodine water purification tablets for use on the contract as follows:

				Date
Type	No. Bottles	Manufacturer	Lot No.	<u>Packed</u>
50-tablet bottles	6,500	Van Brode Corp.	2894-594	8/71
1,000-tablet bottles	900	Van Brode Corp.	2894-1000-1	8/71

The 1,000-tallet bottles were used to fill the blister sheets. The 50-tablet bottles are to be used for comparison tests with the pouched blisters when both are subjected to accelerated environmental storage conditions. These tests are to be performed by CRC and the U. S. Army. CRC will perform the tests on a small number of samples to establish trends, while the U. S. Army is to perform the tests on a more extensive basis so that the statistical results will be of high reliability and confidence.

Evaluation of Tablets

The tablets were analyzed by conducting iodine titration and solubility tests according to the methodology stated in MIL-W-283F. This specification requires the titratable iodine per tablet to be no less than 7.60 mg. The tablet must also dissolve in water at 20°C in less than 5 minutes. The specification stipulates the use of 20-tablet samples for making titration tests and 10-tablet samples for making solubility tests.

THE SECTION OF THE PROPERTY OF

At zero time (i.e., before exposure), the sample quantity consisted of a random selection of ten 1,000-tablet bottles, ten 50-tablet bottles, and 20 pouches (filled from 1,000-tablet bottles). Both titration samples (20 tablets) and solubility samples (10 tablets) were withdrawn from the same bottles. Separate pouches, however, were required for titration and solubility tests to obtain the required 30 tablets. Titration samples (20 tablets) were weighed prior to the titration tests to determine if weight variations were a contributing factor in the dispersion of titration test results.

Table 1 displays the results of the zero time titration tests. The mean value of the titration iodine was calculated according to the relation:

$$I = \frac{\Sigma I}{N}$$

where I is the amount of titratable iodine per tablet in milligrams for each sample of 20 tablets, and N equals the number of samples of a particular type. The mean weight of a 20-tablet sample, W, was calculated at 2239 mg by averaging all 29 test samples*.

The weight biased sample was calculated according to the relation:

 $I_W = \frac{\overline{W}}{W} I$

The values of I_W displayed in Table 1 give an indication of the variation in iodine concentration among the groups of 20 tablets. Assuming a Gaussian or normal distribution, an estimate of the standard deviation was calculated according to:

$$S = \left[\frac{n y(x^2) - (\sum x)^2}{n(n-1)}\right]^{\frac{1}{2}}$$

where:

 $X = I \text{ or } I_W$

Observations made from Table 1 are listed below:

- 1. All samples exceed the minimum requirement of 7.60 mg per tablet.
- 2. The pouched blister sheet samples are of the heaviest average weight and contain the highest average iodine content, while the 50-tablet bottle samples have the lowest average weight and contain the lowest average content. When corrections are made for weight variations, the samples taken from 50-tablet bottles exhibit the highest concentrations of iodine.
- 3. Pouched blister sheets have the highest average weight, which seems contrary to logic since they would be expected to have lost some weight during the filling and sealing operation. It is concluded that the filling and sealing operation imposes negligible wear or degradation to tablets, and that their high average weight is due to their being filled from a few 1,000-tablet bottles containing tablets of greater than average size.
- 4. The tablets from 50-tablet and 1,000 tablet bottles seem, in general, to be identical, since the differences between mean values of titratable iodine content are less than 1/2%. A larger sampling quantity would probably reduce the statistical difference even further.

^{*}One sample weighing was overlooked in titration tests.

TABLE 1 - Zero Time Exposure Titration Tests Daca

	Sample	W (mg per	Ī (mg	Ī (mg
Sample Type	No.	20 tablets		per tablet)
			**************	***************************************
50-Tablet Bottles	1	2160	8.19	8.49
	2	2220	8.12	8.19
•	3	2230	8.31	8.34
	4	2190	8.12	8.30
•	5	2160	8.06	8.35
	6	2210	8.25	8.36
	7		8.25	
	8	2180	8.19	8.41
	9	2190	8.06	8.24
	10	2160	<u>8,06</u>	<u>8.35</u>
			01:	
		Ţ.	=8.16, S= 0.	16 $\bar{I}_{w} = 8.34, S = 0.11$
1,000 Tablet Bottles	1	2330	8.06	7.75
	2	2200	8.12	8.26
	3	2340	8.69	8.31
	4	2260	8.12	8.04
		2250	8.12	8,08
	5 6`	2230	8.12	8.15
	7	2190	8.25	8.43
	8	2250	8.25	8.21
•	9	2180	8.06	8.28
	10	2230	8.19	8.22
		T= 8	3.20, S=0.18	Ī=8.17, S=0.19
Pouched Blister Sheets				
rodened blister Sneets		2270	8.31	8.20
	2 3	2260	8.31	8.23
	_	2270	8.44	8.32
	4	2270 2280	8.31	8.20
	5 6	2280	8.25	8.10
	7	2310	8.44 8.50	8.29
	8	2290	8.25	8.24
	9	2280	8.44	8.07
	10	2280		8.29 8.16
			<u>8.31</u>	<u>8.16</u>
		T= W=2 239	8.36, S=0.09	9 T _w =8.21, S=0.08

Table 2 presents the results of the zero time solubility tests. The recorded times indicate the period required to dissolve the 10-tablet samples in a graduated cylinder containing 250 ml of distilled water at 20°C. The following observations are made concerning Table 2:

- 1. All 50-tablet and 1,000-tablet bottle samples pass the solubility test requirement to dissolve in less than 5 minutes.

 Two samples among the pouched blister sheets, however, exceed the maximum time of 5 minutes for dissolving.
- 2. Individual samples exhibited large variations in their dissolving rate. The mean dissolving times, however, for 50-tablet and 1,000-tablet bottles are approximately equal, while the average of the pouched blister sheets took approximately 32 seconds longer. The longer period to dissolve the tablets in the pouched blister sheets is probably caused by their generally large size as indicated in Table 1. It is also possible that the increased solubility time for these tablets is caused by their second exposure to air during the filling and sealing operations.

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Blister Sheet Seal Leakage Tests

Fifty samples of filled and sealed blister sheets containing one dozen tablets each were selected and subjected to vacuum leakage tests to determine the integrity of seals made with the modified sealing tray. These tests consisted of submerging the blister sheets in a beaker of water within a bell jar which was then cycled twice between atmospheric pressure and a differential vacuum corresponding to 27 inches of mercury. This vacuum was maintained for 30 minutes on the first cycle and 2 hours on the second cycle, and was a far more severe test than the 20 inches of mercury vacuum differential and 5-minute cycle period called for in the contract. A seal that leaked would have been visually indicated by wetted iodine tablets which would have changed from a gray to a brown color.

Examination of the samples after testing showed that none of the seals leaked—a very encouraging sign in view of the severity of the tests. It should be noted, however, that variations in the sealing parameters of heating time, cooling time, and sealing pressure may occur in production as the result of residual heat buildup in the sealing tray. These parameters then may have to be adjusted to insure that quality seals are being maintained.

TABLE 2 - Zero Time Exposure Solubility Test Data

Sample Type	Sample	Average Time per 10-tablet sample Min -Sec
<u>Dump20 1,700</u>		
50-Tablet Bottles	1	2 - 22
	2	3 - 47
	3	
	4	3 - 07
	5	3 - 36
•	6	3 - 40
	7	3 - 38
	8	3 - 14
	9	4 - 32
	10	3 - 32
		$\bar{T} = 3 - 29$, $S = 35 \text{ sec.}^{\prime}$
1,000-Tablet Bottles	1	3 - 33
2,000 ,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,	2	4 - 45
	3	2 - 38
	3 4	3 - 29
	5	3 - 34
	5 6	3 - 13
	7	2 - 49
	8 -	3 - 56
	9	2 - 59
	10	3 - 06
		$\bar{T} = 3 - 26$, $S = 35 \text{ sec.}$
Pouches	1	2 - 59
	2	3 - 36
	3	4 - 50
	4	4 - 57
	5	3 - 20
	6	6 - 21
	6 7 8	4 - 43
	8	2 - 35
	9	3 - 31
	10	5 - 06
		$\bar{T} = 4 - 00$, S = 74 sec.

The nature and cause of typical seal imperfections and method of remedy are described below.

Imperfection	Cause	Remedy
Irregular and distorted seals and blister sheets.	Excessive sealing heat.	Decrease sealing time.
Weak seals.	Insufficient seal- ing heat.	Increase sealing time.
Combination of weak and strong seals.	Uneven pressure distribution over tray.	Adjustment of lower tray legs.
Localized burns in seals.	Pressure sensitive teflon sheet on sealing tray worn out.	Replace teflon sheet.
Non-uniformity of seals.	Insufficient cooling time or excessive heat buildup in sealing tray.	Increase cooling rate; stop production until residual heat dissipates from sealing trays.

ACCELERATED STORAGE TESTS

Description of Tests

A sample number of the pouched blister sheets and 50-tablet bottles were introduced into two environmental chambers simulating accelerated storage conditions. One chamber generated an environment of 100°F at 95 percent RH, while the second chamber generated an environment of 140°F at ambient absolute humidity. Approximately 100 pouches and fifty 50-tablet bottles were placed in each chamber at the commencement of tests. At the end of 1 week, 1 month, and 2 months, samples of the 50-tablet bottles and pouches were withdrawn from each chamber and subjected to the iodine titration and solubility tests which have been described. The results of these tests are described below.

Results of Tests

Tables 3, 4, and 5 tabulate the results of titration and solubility tests of samples subjected to 1-week, 1-month, and 2-month storage at 100°F and 95% RH. Results and conclusions regarding these tests are listed below:

TABLE 3 - One Week Exposure at 100°F and 95 Percent RH Titration and Solubility Test Data

Sample Type	Sample No.	<u>I (mg)</u>	Sample Type	Sample No.	Time (<u>Min - Sec</u>)
Pouched	1	7.93	Pouched Blister	1	4 - 59
Blister	2	8.31	Sheets	1 2	6 - 22
Sheets		8.50		3	6 - 07
	4	8.38		4	4 - 56
	· 5	8.12		5	5 - 20
	6	8.12		6	4 - 26
	7	8.19		7	5 - 38
	8	8.19		8	5 57
	9	8.25		9	5 - 22
	10	<u>8.38</u>	•	10	<u>7 - 31</u>
	Ī=8.24	S= 0,16		T =5	-40, S= 0-53
50-Tablet	1	8.38	50-Tablet	1	3 - 51
Bottles	2	8.12	Bottles	2	3 - 45
	3	8.12		3	3 - 31
	4	8.06		4	3 - 50
	5	7.87		5	3 - 30
	F	8.57		6	3 - 55
	7	8.31		7	3 - 33
	8	8.12		8	4 - 28
	9 .	8.19		9	4 ~ 35
	10	8.50		10	3 - 25
	Ī=8.22,	S= 0.21		Τ=3	-50, S= 0-24

TABLE 4 - ONE MONTH EXPOSURE AT 100°F AND 95 PERCENT RH TITRATION AND SOLUBILITY TEST DATA

	Sample		1	Sample	Time
Sample Type	No.	I(mq)	Sample Type	No.	(Min - Sec)
	_			_	4 40
Pouched	1	8.06	Pouched	1	4 - 40
Blister	2	8.12	Blister	2	5 - 21
Sheets	3	8.31	Sheets	3	3 - 39
	4	8.31		4	6 - 54
•	5	8.25		5	4 - 46
	6	8.31		6	5 - 57
	7	8.19		7	7 - 32
	8	8,25		8	5 - 12
	9	8.19		9	7 - 10
	10	7.42		10	6 - 44
			:.		
	T=8.14 S=	0.28		T ≈5-	48 S= 1-16
20 m-1-1-4	•	0.57	50-Tablet	1	4 - 36
50-Tablet	1 2	8.57		2	4 - 05
Boutles		8.12	Bottles	3	4 - 03
	3	7.99			
	4	8.57		4	3 - 33
	5	8.12		5	4 - 32
	6	8.06		6	3 - 42
	7	8.69		7	3 - 45
	8	8.69		8	4 - 15
	9	8.05		9	3 - 23
	10	8.05		10	2 - 40
	T=8.29 S=	0.29		<u>T</u> =3.	-51 S= C-36

TABLE 5 - TWO MONTH EXPOSURE AT 100°F AND 95 PERCENT RH TITRATION AND SOLUBILITY TEST DATA

	Sample	1		Sample	Time
Sample Type	No.	I (mg)	Sample Type	No.	(Min - Sec)
Pouched	1	7.86	Pouched	1	5 - 50
Blister	3	8.00	Blister	2	5 - 02
Sheets	3	8.31	Sheets	3	3 - 58
	4	8.06		4	5 - 37
	5	8.50		5	3 - 55
	6	8.00		6	4 - 30
	7	7.81		7	6 - 50
	8	7.81		8	6 - 04
	9	7.93		9	5 - 43
•	10	7.93		10	4 - 40
	T= 8.02,	S= 0.22		T= 4.	-55, S= 0-28
50-Tablet	1	8.12	50-Tablet	1	5 - 01
Bottles	2	7.93	Bottles	2	5 - 58
	3	7.48		3	6 - 17
	4	8.37		4	4 - 50
	5	7.61			
	6	8.18			
	T=7. 95,	S= 0.35		T =	5-16, S= 0.29

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1. Storage at 100°F at 95% RF does .ot significantly affect the titratable iodine content in tablets in either the 50-tablet bottles or the pouched blister sheets.

- 2. Tablet solubility deteriorates during storage. After 1-week storage, most of the samples from pouched blister sheets require more than 5 minutes to dissolve. After 1 week, however, there is no further deterioration in the solubility. The 50-tablet bottle samples, however, indicate little deterioration in solubility after 1-week and 1-month storage, but deterioration in solubility is pronounced after 2-month storage. The deterioration in tablet solubility is about equal for both the 50-tablet bottles and the pouched blister sheets after 2-month storage.
- 3. The high estimate of the standard deviation among solubility samples indicates that a higher sample quantity is required for more accurate statistical results.

Tables 6 and 7 display the results of titration and solubility tests for samples subjected to 1-week and 1-month storage at 140°F. Unfortunately, there are no quantitative 2-month test results, since between the 1 and 2 month period, the chamber malfunctioned and subjected the samples to temperatures up to 180°F. Comments regarding these tables are listed below:

1. A loss in titratable iodine results in both the pouched blister sheets and 50-tablet bottles. The deterioration, however, is much more severe for the 50-tablet bottles after 1-month tests where the titratable iodine equals 5.00 mg, as compared to 7.81 mg for the blister sheets. The more rapid deterioration of the bottled tablets is attributed to iodine vapor loss around the bottle cap threads.

- 2. Both 50-tablet bottles and pouched blister sheet samples failed the solubility tests. Generally, the solubility time of the 50-tablet bottle is longer than the pouched blister sheets.
- 3. Based on the above results, it is concluded that the pouched blister sheet will afford better protection of the iodine water purification tablet than the present 50-tablet bottle when stored at elevated temperature:.

TABLE 6 - One Week Exposure at 140°F - Titration and Solubility Test Data

	Sample			Sample	e Time
Sample Type	No.	I (mq)	Sample Type	No.	(Min - Sec)
				•	2 22
Pouched	1	7.81	Pouched	1	3 - 38
Blister	2	7.96	Blister Shee		6 - 13
Sheets	3	7.96		3	4 - 19
	. 4	8.19		4	4 - 04
	5	7.87		5	3 - 31
	6	8.19		6	6 - 55
	7	7.81		7	3 - 59
	8	7.74		8	8 - 06
	9	8.31		9	5 - 09
	10	<u>7.81</u>		10	<u>7 - 23</u>
3	Ē=7.97	S= 0.20			T=5-20, S= 1-42
50-Tablet	1	7.68	50-Tablet	1	5 - 13
Bottles	2	8.06	Bottles	1 2	5 - 45
	3	7.42		3	4 - 59
	4	7.93		4	5 - 26
	5	7.74		5	5 - 45
	6	7.93		6	4 - 35
	7	7.87		7	9 - 26
	8	8.06		8	5 - 33
	9	7.68		9	6 - 29
	10	-		10	<u>5 - 56</u>
	70	7.74	1	70	3 - 30
	Ī=7.81,	S= 0.20	I		T=5-55, S= 1-20

TABLE 7 - ONE MONTH EXPOSURE AT 140°F - TITRATION
AND SOLUBILITY TEST DATA

Sample Type Pouched Blister Sheets	No. 1 2 3 4 5 6 7 8 9 10	I (mg) 7.62 8.12 7.81 7.62 7.62 7.62 7.74 7.87 7.93 7.93 7.93	Sample Type Pouched Blister Sheets	No. 1 2 3 4 5 6 7 8 9 10	Time (Min - Sec) 6 - 1.2 5 - 14 7 - 31 6 - 42 8 - 55 8 - 35 7 - 33 6 - 05 5 - 02 5 - 24
	Ī=7.81 S	= 0.16		T =6	-42 S= 1-23
50-Tablet Bottles	1 2 3 4 5 6 7 8 9 10	6.09 4.06 5.08 5.71 5.14 5.39 3.99 4.25 5.07 5.20	50-Tablet Bottles	1 2 3 4 5 6 7 8 9 10	6 - 15 9 - 23 9 - 15 6 - 52 7 - 46 9 - 02 4 - 49 9 - 28 5 - 53 6 - 42
		0.70		T=7-	-33 S= 1-41

PILOT PRODUCTION OPERATION

Pilot Production Summary

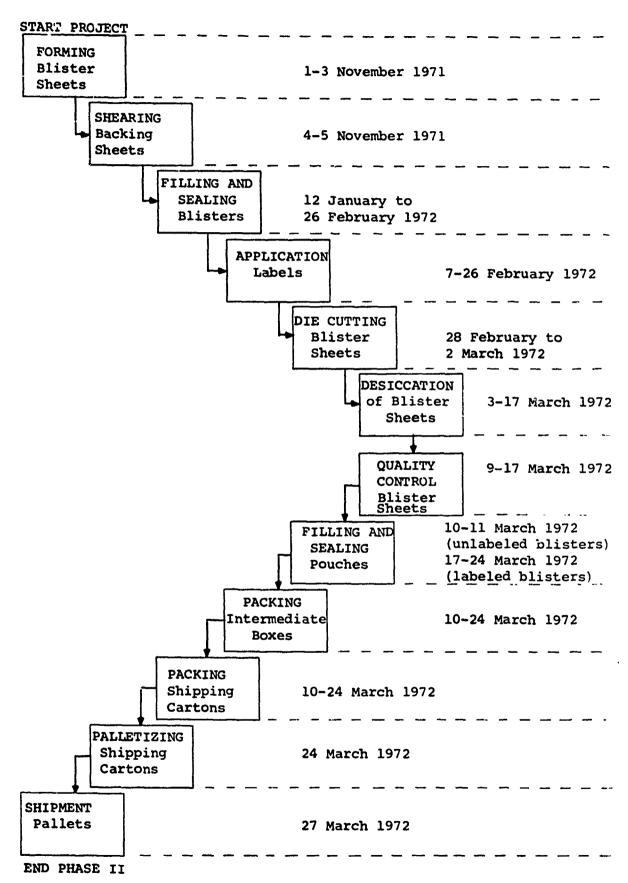
Figure 5 shows a flc. chart of the sequence of pilot production processes and their dates of occurrence. The pilot production processes include, in chronological order, the following operations:

1. Forming from rolls of 5 mil Aclar 22A film, twelve thousand (12,000*) sheets of blisters measuring 2½" x 13".

- 2. Shearing from rolls of 5 mil Aclar 22A film, twelve thousand (12,000*) backing sheets measuring 2½" x 13".
- 3. Filling and sealing eight hundred thousand (800,000) tablets in the blister and backing sheets produced.
- 4. Applying nine thousand five hundred (9,500) labels to the sealed blister sheets.
- 5. Die cutting the labeled and unlabeled blister sheets to make an excess of sixty thousand (60,000) blister sheets containing twelve (12) tablets each.
- 6. Desiccation of the sixty thousand (60,000) blister sheets in lots of two thousand (2,000) blister sheets each.
- 7. Hand filling and sealing in pouches sixty thousand (60,000) blister sheets in thirty thousand (30,000) pouches.
- 8. Erecting and filling three thousand (3,000) intermediate boxes, each containing ten (10) pouches.
- 9. Erecting, filling, and stapling one hundred fifty (150) shipping cartons each containing twenty (20) intermediate boxes.

The pilot production operation covered a 5-month period from 1 November 1971 until 27 March 1972. It should be recognized that this was not a continuous operation. There was an interruption of

^{*}Excessive quantities were produced to account for production rejects.



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FIGURE 5. PILOT PRODUCTION PROCESSES AND DATES

2 months allowed between the fabrication of the blister and backing sheets and the filling and sealing of blisters to permit Phase I tests to be completed and evaluated. This period was deemed necessary to assure that the manufacturing processes would produce a package of good quality. In addition, production operations were ceased during the desiccation of blister sheets and quality control tests. Basically the same types of machinery were used in fabrication of the improved package as were used in the original pilot production. A description of how this machinery operates has been given in detail in Reference 1, and therefore will not be repeated in this report. The pertinent adjustments of machine parameters needed to produce the improved package, however, are given quantitatively where applicable for each manufacturing process.

Blister Forming

Blisters from Aclar 22A film were formed on a Packaging Industries Sentinel Model JF-14 Blister Forming Machine. The machine settings were as follows:

Heater Temperature - 800°F outer elements; 600°F center elements

Cycle Period - 4.1 seconds

Forming Period - 2.5 seconds

Cycle Increment - 2.5 inches

Twelve thousand six hundred cycles were made with this machine over a 2½-day period. The manpower requirements consisted of a machine operator and one production worker. Approximately 5% of the blisters were malformed because of improper machine settings so that approximately 12,000 usable blister sheets resulted. Figure 6 shows the packing list for the Aclar 22A film which was used to make the blister sheets as well as the backing sheets. During production, roll #G808-124-3-1 did not form well and was taken off the machine and used for shearing the backing sheets.

Backing Sheet Shearing

The same Packaging Industries Sentinel Model JF-14 was used as a shearer to cut the backing sheets into $2\frac{1}{2}$ " x 13" sheets from

FABRICATED PRODUCTS DIVISION

ALLIED CHEMICAL CORPORATION

P. O. BOX 697 POTTSVILLE, PENNA. 17901

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ignee (Order No. 10	914			Date	*************************	
	£			Shipped	Via	********	
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		FILM PA					
TYPE	GAUGE	ROLL NUMBER	LENGTH	GROSS	TARE	NET	WIDT-i
29 A	5-MIL	G-08124-3-2	1000'	149.0	1810	65.0	141/2
IL.	11	Gfc8124-3-3	1000			66.0	11
/(11	G80x 124-3-1	1000	141.0	18.0	66.0	"
10	11	GNF 123-3-2	500		1 .	57.0	11
	"(680F 123-3-3	1025	167-0	18.0	174.0	"
11	"	GFUS 123 - 3-1	1620			7510	1
						<u> </u>	1
							<u> </u>
	. /	PALECT		50.0			<u> </u>
		CART ONS			54.0	<u> </u>	
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FIGURE 6. ACLAR FILM PACKING LIST

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14½" wide rolls of Aclar 22A. In this situation, the heater was turned off and the forming mold disengaged. The cycle rate during shearing was 3 seconds, which was the maximum speed for this machine.

Filling and Sealing Iodine Tablets in Blister Sheets

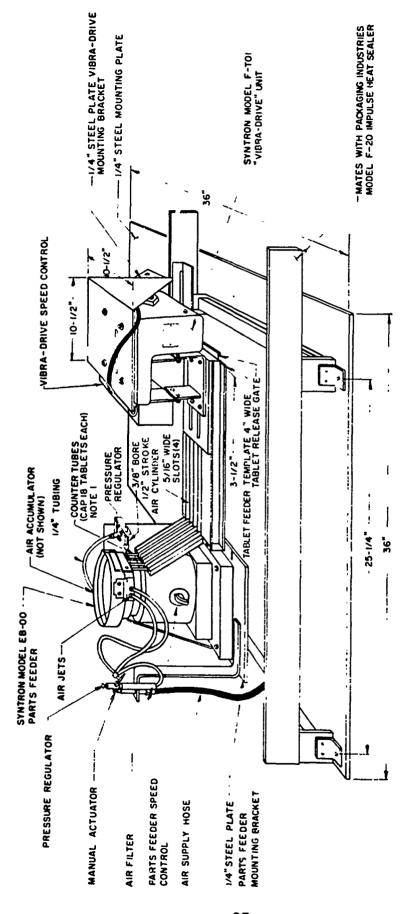
Figure 7 shows a schematic of the tablet filling apparatus which was designed by Columbia Research Corporation for integration with the PI Model F-20 Impulse Heat Sealer. This equipment was used to fill and seal the iodine tablets in the blister sheets. For each cycle of operation, this combination of equipment filled and sealed 72 iodine tablets between a $2\frac{1}{2}$ " x 13" blister sheet and a $2\frac{1}{2}$ " x 13" backing sheet.

During the production operation, the F-20 settings were adjusted as follows:

- 1. Heating Period 2.5 to 3 seconds
- 2. Cooling Period 15 seconds
- 3. Platen Pressure 80 psi
- 4. Pressure Switch Setting 75 psi
- 5. Heating Power 50 volts

Other methods which were employed to produce uniform blister sheet seals were the following: a fan was used to cool the upper tray to dissipate the heat during production. To preclude degradation of the tablets due to moist air, the pressurized air in contact with the tablets in the filling apparatus was predried by passing through three separate filtering systems. The legs on the lower sealing tray were adjusted as necessary to apply the proper local pressure to counteract tray warpage that occurred during production operation.

Prior to filling and sealing blister sheets, five cycles were made on the machine to stabilize the heat in the sealing tray. The filling and sealing operation was performed over a 6½-week period between 13 January and 28 February 1972. In operation, blister sheets were filled and sealed at a rate of one every 25 seconds. All blister sheets were contained in an insulated room kept at low temperature and low humidity prior to their being filled and sealed. The humidity and temperature in the room were



NOTE ! COUNTER TUBES HINGED TO FEEDER BOWL

FIGURE 7. AUTOMATIC TABLET FILLER

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checked each morning and afternoon with a psychrometer. Table 8 shows a daily log of the psychrometer readings and production rate, while Figure 8 is a psychrometer chart indicating the range the readings covered over this period. In all instances, the humidity was within the 55 grains per pound of dry air limit. Table 8 indicates that for most of the production days the actual absolute humidity was approximately half this value.

During the filling and sealing operation period, 16 of the heating elements broke and were replaced. Fourteen of these elements failed on the lower tray, the other two failures occurring on the upper tray. Failure of these elements was attributed to the cold working of the heating elements during the removal of the Aclar blister sheets from the lower tray. The Aclar blister sheets exhibited a strong adhesion to the tray in the sealing area which may be attributed to the fact that Aclar and teflon belong to the same generic family of fluoroplastics. In addition, teflon sheeting had to be replaced approximately every 1,000 cycles due to excessive wear caused by the removal of the Aclar sheets after sealing. Also, whenever a heating element broke, it created a hot spot which burned through the teflon sheeting. A more effective coating on the sealing tray which would be less adhesive to the Aclar sheets would be expected to eliminate these problems. At this time, however, there is no commercially available material that provides a better release of the Aclar film than the present teflow coated glass sheet.

Labeling Blister Sheets

Special labels were fabricated with the color match and use instructions for the blister sheets. The remaining blister sheets were left unlabeled so that a small quantity of packets could be made without the labels to assess the quality of this package in case the water containment in the label continued to present a problem.

Die Cutting Blister Sheets

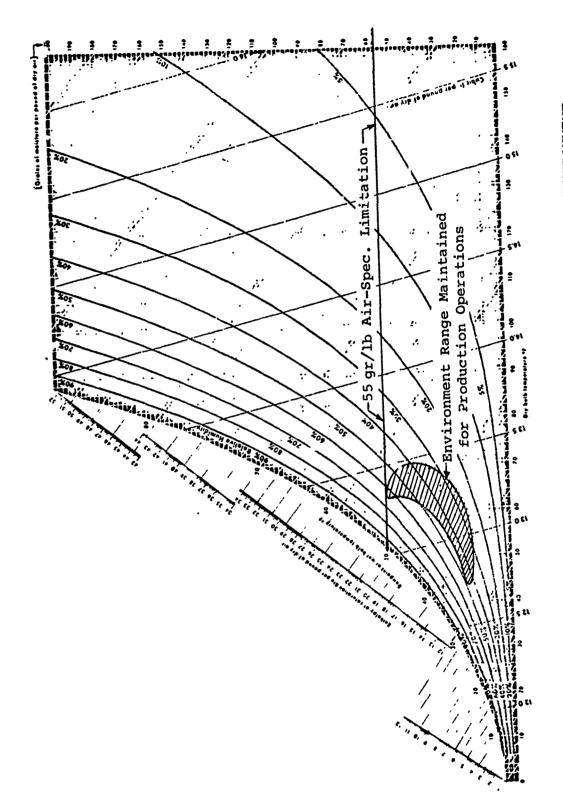
The labeled and unlabeled blister sheets were die cut on an automatic die cutting press. Two workers--one machine operator and a production worker--were required to cut the sheets. All 12,000 sheets were cut over a 3 1/2-day period. This operation went very smoothly and was accomplished without major difficulty of any type.

DATE 11ME	DRY BULB TEMP DEG (F)	WET BULB TEMP DEG (F)	ABSOLUTE HUMIDITY GRAINS OF / LB OF H20 / DRY AIR	QUANTITY (1000 TABLET (BOTTLES)
12 Jan 1972 AM/PM	65/	57/	55/	21
14 Jan AM∕PM	65/57	57/47	30/	20
17 Jan AM/PM	47/	36/	19/	25
18 Jan AM/PM	60.5/	46/	24/	31
19 Jan AM/PM	65.5/	51.5/	34/	15
20 Jan AM/PM	61/64	47/50	26/31	27
21 Jan AM/PM	65/66	_53/53	40/39	26
24 Jan Am/PM	65/68	53/55	40/44	15
25 Jan AM/PM	64/59	51/45	35/22	25
26 Jan AM/PM	44/56	34/43	13/21	21
27 Jan AM/PM	57/60	46/47	29/27	32
28 Jan AM/PM	64/65	50/49.5	31/28	22
31 Jan AM/PM	54/59	40/45	14/22	32
l Feb AM/PM	58/63	44/48	20/26	25
am56h	64/64	49/50	27/31	26
3 Feb AM/PM	64/64	50/51	31/35	34
4 Feb AM/PM	51/58	40/45	19/24	25
7 Feb AM/PM	65/	48/	23/	15
8 Feb AM/PM	53/60	39/44	13/17	25
9 Feb AM/PM	60/60	45/45	20/20	25
10 Feb AM/PM	61/63	45/46	19/20	28
11 Feb AM/PM	62/62	46/46	21/21	36
14 Feb AM/PM	60/63	47/49	27/29	19
15 Feb AM/PM	61/65	47/50	26/28	25
16 Feb AM/PM	52/57	42/43	23/19	14
18 Feb AM/PM	63/64	49/50	28/31	31
AM/FRb	/60	/47	/27	8

TABLE 8. DAILY LOG OF ENVIRONMENT AND PRODUCTION DURING TABLET FILLING AND SEALING OPERATIONS

DATE TIME	DRY BULB TEMP DEG (F)	WET BULB TEMP DEG (F)	ABSOLUTE FUMIDITY GRAINS OF / LB OF H20 / DRY AIR	QUANTITY (1000 TABLET (BOTTLES)
21 Feb AM/?M	59/63	45/48	22/26	22
22 Feb AM/PM	64/64	48/48	24/24	22
AM/PM 23 Feb AM/PM	56/56	43/43	21/21	35
24 Feb	69/69	53/53	35/35	30
25 Feb AM/PM	68/68	53/53	36/36	29
26 Feb AM/PM	65/	50/	28/	14
	1			
	<u> </u>			
<u></u>	<u> </u>			
	<u></u>		<u> </u>	

TABLE 8. DAILY LOG OF ENVIRONMENT AND PRODUCTION DURING TABLET FILLING AND SEALING OPERATIONS (cont.)



PSYCHROMETRIC CHART INDICATING FILLING AND SEALING ENVIRONMENT FIGURE 8.

Desicrating and Evaluation of Cut Blister Sheets

The labeled blister sheets were grouped in lots of approximately 2,000 each and filled in 21 polyethylene bags. Five pounds of anhydrous calcium sulfate desiccant were added to each bag, and the bags then heat sealed shut. The desiccant was used to remove any residual moisture which may have been entrapped within the labels prior to pouching. This operation had proven effective in earlier tests to eliminate the moisture in the labels which had been a problem on the previous pilot production of the package. The blister sheets were kept in these bags for 1 week. At the end of 1 week, six blister sheets from each of the 21 bags were removed and sealed in three pouches, and the polyethylene bags immediately resealed. The samples were identified and then placed in an environmental chamber set at 140°F. In addition, five 50-tablet bottles were placed in this chamber as a control.

At the end of 1 week, one pouch was opened from each lot plus one 50-tablet bottle to determine if any discoloration of the tablets had occurred. The pouched samples displayed insignificant discoloration of the tablets while the 50-tablet bottle sample contained tablets which were severely discolored. It was concluded then that all of the blister sheets within their polyethylene bags were adequately dried and were ready for pouching. It is believed that the desiccation procedure plus the fact that the labeling operation was performed in a low humidity environment has eliminated the possibility of tablet deterioration by moisture entrainment over long range testing. The remaining two samples from each lot will remain in the test chamber for approximately three additional months to assess any further long term effects.

Pouching of Blister Sheets

Figure 9 shows a schematic of the heat sealer with a pouch sealer guide for this particular production operation. The sealer was set at a temperature of 425°F and the pressure adjusted such that the seals would separate and tear at approximately the same time when pulled to destruction. If the pressure becomes excessive or the heat too high, the material tears at the edge of the seal when tested. If the pressure or heat is too low, the seal separates at too low a tension. The seals of pouches were checked periodically to assure the proper pressure and temperature were being maintained.

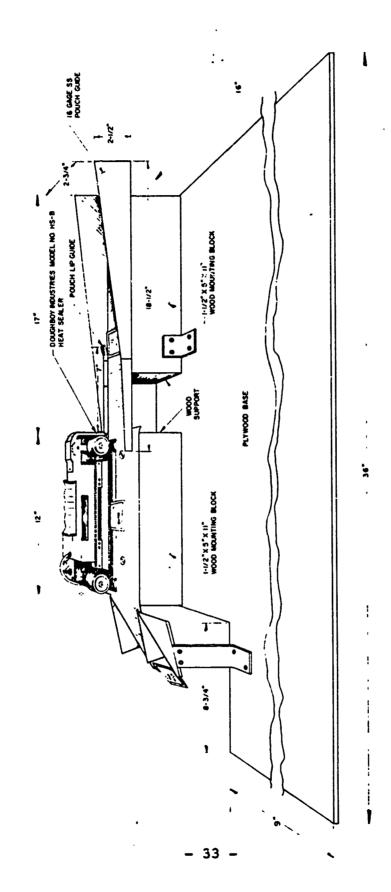


FIGURE 9. POUCH SEALER

Packing Pouches in Intermediate Boxes and Shipping Cartons

Intermediate boxes and shipping cartons were supplied by L. Gordon and Son, Inc. The 3,000 intermediate boxes were of paperboard and measured 3-1/2" x 3-1/2" x 2". Each box was designed to contain ten filled pouches and three 50-tablet bottles. A paperboard insert within this box gave a snug fit to the pouches and bottles.

The 150 shipping cartons were fabricated from a weather resistant, single wall, corrugated fiberboard of type V3C conforming to Federal Specification No. PPP-B-636E. These cartons measured 10-3/4" x 7-1/2" x 7-3/4" and held 20 intermediate boxes. A heavy duty manually operated stapler was used to erect and seal the shipping carto's.

Packaging of intermediate boxes and shipping cartons was integrated with the operation of sealing the blister sheets in pouches at Columbia Research between 10 March 1972 and 24 March 1972. Approximately two man-days were required to hand pack the 30,000 pouches and the 6,500 50-tablet bottles into the intermediate boxes and the intermediate boxes into shipping cartons.

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CONCLUSIONS AND RECOMMENDATIONS

- 1. The present improved flexible package for the iodine water purification tablet affords better protection to the tablet than the present 50-tablet bottle when stored at elevated temperature. Other advantages of the flexible package over the bottle are:
 - a. Each tablet is individually sealed.
 - b. Any deterioration of the tablets can be easily detected visually.
 - c. The pouch has a convenient flat shape.
 - d. The total weight of the flexible package of 24 tablets is 1/3 that of the 50 tablet bottle used previously.
 - e. The directions for use are much more legible and informative.

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- 2. The following major improvements have been made to the flexible package for iodine water purification tablets on this contract:
 - a. Removal of entrapped moisture from the blister sheet label by a desiccation process.
 - b. Improved blister sheet and pouch heat seals.
 - c. A reduction in cost of the package by approximately 25 percent. This reduction has been primarily realized in the more efficient operation of producing Aclar blister sheets.
- 3. Given present material costs, it is estimated that the flexible package with 24 iodine water purification tablets could be mass produced for 15 to 20 cents each of which 6 cents would represent the cost of the tablets.
- 4. A study should be undertaken to determine whether the package cost could be further reduced through less expensive packaging materials. It is expected that substitution of the present pouch film with several of the more popular commercially available types, plus development of a cheap paper label with gray adhesive for the blister could effect considerable cost savings to the package. In addition, because of the expense of Aclar film in blister sheets, the use of thinner gages should be investigated.

REFERENCES

- Brown, D. F. and C. T. Derick, "A Blister Sheet and Pouch Overwrapper Package for the Iodine Water Purification Tablet," U. S. Army Land Warfare Laboratory Technical Report No. LWL-CR-05S69 (October 1971).
 - 2. Pioneering Research Laboratory Lab Report No. 70-F-S (4 March 1971).